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मानक

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IS 1528-9 (2007): Methods of Sampling and Physical Tests for Refractory Materials, Part 9: Determination of True Density [MTD 15: Refractories]



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“Knowledge is such a treasure which cannot be stolen”



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भारतीय मानक

उष्मासह सामग्रियों के नमूने लेने की और भौतिक परीक्षण पद्धतियाँ

भाग 9 वास्तविक घनत्व ज्ञात करना

( चौथा पुनरीक्षण )

*Indian Standard*

**METHODS OF SAMPLING AND PHYSICAL TESTS  
FOR REFRACTORY MATERIALS**

**PART 9 DETERMINATION OF TRUE DENSITY**

*( Fourth Revision )*

ICS 81.080

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**BUREAU OF INDIAN STANDARDS**  
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## NATIONAL FOREWORD

This Indian Standard (Part 9) (Fourth Revision) which is identical with ISO 5018 : 1983 'Refractory materials — Determination of true density' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Refractories Sectional Committee and approval of the Metallurgical Engineering Division Council.

This standard was originally published in 1962 and subsequently revised in 1974, 1980 and 1995. This revision of the standard has been taken up to align it with ISO 5018 : 1983 by adoption, under dual numbering system.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.
- b) Comma (,) has been used as a decimal marker, while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted in their places, are listed below along with their degree of equivalence for the editions indicated:

<i>International Standard</i>	<i>Corresponding Indian Standard</i>	<i>Degree of Equivalence</i>
ISO 565 : 1990 Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures	IS 1528 (Part 14) : 1974 Methods of sampling and physical tests for refractory materials: Part 14 Determination of sieve analysis ( <i>first revision</i> )	Technically Equivalent
ISO/R 836 : 2001 Vocabulary for the refractories industry	IS 4041 : 2006 Glossary of terms relating to refractory materials ( <i>second revision</i> )	Identical
ISO 5022 : 1979 Shaped refractory products — Sampling and acceptance	IS 1528 (Part 7) : 1974 Methods of sampling and physical tests for refractory materials: Part 7 Methods of sampling and criteria for conformity ( <i>first revision</i> )	Technically Equivalent

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard*  
**METHODS OF SAMPLING AND PHYSICAL TESTS  
FOR REFRACTORY MATERIALS**

**PART 9 DETERMINATION OF TRUE DENSITY**

*( Fourth Revision )*

**1 Scope and field of application**

This International Standard establishes a method for measuring the true density of refractory products and raw materials.

**2 References**

ISO 565, *Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures.*

ISO/R 836, *Vocabulary for the refractories industry.*

ISO 5022, *Shaped refractory products — Sampling and acceptance.*

**3 Definitions**

For the purpose of this International Standard, the following definitions apply.

**3.1 true density :** The ratio of the mass of a quantity of dried material to its true volume.

**3.2 true volume :** The volume of the solid material in a porous body.

**4 Principle**

**4.1** The true density is determined by measuring the dry mass and the true volume of a sample of the material after it has been crushed and ground to such a particle size that as far as possible no closed pores remain. The volume of the ground material is determined using a pycnometer and a liquid of known density, the temperature of the liquid being controlled or carefully measured.

**4.2** Unfired refractory products and basic products may require pre-treatment, the conditions of which shall be agreed between the parties concerned.

**5 Apparatus**

**5.1 Pycnometer,** of capacity from 25, 50 or 100 ml, fitted with a ground stopper having a capillary bore.

**5.2 Balance,** with an accuracy of  $\pm 0,1$  mg.

**5.3 Vacuum equipment,** capable of reducing the pressure to a value not greater than 25 mbar<sup>1)</sup>, with a means of measuring the pressure.

**5.4 Thermostatically controlled bath,** capable of being maintained at a temperature from 2 to 5 °C above the ambient temperature with an accuracy<sup>2)</sup> of  $\pm 0,2$  K.

**5.5 Test sieve,** 63  $\mu\text{m}$  aperture complying with the requirements of ISO 565.

**5.6 Drying oven.**

**5.7 Desiccator.**

**6 Test material**

**6.1** The samples to be measured shall be selected in accordance with ISO 5022<sup>3)</sup> or another standard sampling plan agreed by the interested parties.

**6.2** In the case of a shaped refractory product, the number of test pieces to be tested for each item shall be agreed between the interested parties and shall be stated in the test report. To facilitate statistical evaluation when several bricks are tested, the same number of test pieces shall be taken from each brick.

**6.3** The test material (test piece) shall be crushed and ground to pass completely through the test sieve (5.5).

1) 1 bar =  $10^5$  Pa

2) This accuracy of temperature control is necessary because the method is very sensitive to variations in temperature. Because of the different coefficients of thermal expansion of the vessel and of the liquid, significant errors arise if there are variations in the temperature.

3) Sampling of unshaped refractory products and primary materials will form the object of a future International Standard.

**6.4** Care shall be taken that the crushing and grinding operations do not introduce foreign matter or moisture into the material.

**6.5** Before the test, the materials to be tested shall be dried at  $110 \pm 5$  °C to constant mass, that is to say until two successive weighings made before and after at least 2 h in the drying oven (5.6) do not differ by more than 0,1 % of the mass of the test material. Before each weighing, the test material shall be placed in the desiccator (5.7) until it has cooled to room temperature.

**6.6** Care shall be taken during the preparation of basic refractory materials to prevent any hydration. It is permissible that these materials should be dried at 500 °C; if this is done, the fact shall be stated in the test report.

## 7 Procedure

### 7.1 Determination of the initial mass of test material

**7.1.1** Clean the empty pycnometer (5.1) and ensure that it is perfectly dry. It is recommended that leather fingers be used to manipulate the pycnometer. Allow it to come to a temperature near to the ambient temperature.

**7.1.2** Weigh the cleaned and empty pycnometer with its stopper in position to the nearest 0,000 2 g

**7.1.3** Introduce into the pycnometer a quantity of the dry test material equal to approximately 1/3 of the volume of the pycnometer. When the pycnometer and its contents have again come to the ambient temperature, weigh it to the nearest 0,000 2 g. The difference in the two weighings is the initial mass of the test material ( $m_1$ ).

NOTE — For an alternative procedure which may be used if the test material is difficult to wet with the liquid (see the annex).

### 7.2 Determination of the mass of the pycnometer filled with a quantity of the test material and with test liquid

**7.2.1** Add to the pycnometer (weighed in accordance with 7.1.3) a quantity of deaerated boiled water or another liquid of known density (see the table), so that the pycnometer is filled to 1/2 or 2/3 of its capacity. Place the pycnometer in a desiccator and expose it to a vacuum (see 5.3) whose pressure is not greater than 25 mbar until no more air bubbles are seen to rise. The pycnometer may be shaken by means of a shaking device mounted in the desiccator, or by some other method, to ensure complete wetting. When a liquid other than water is used, care shall be taken to ensure that it does not boil under the pressure used.

**7.2.2** Fill the pycnometer almost completely with water or with the other chosen liquid and allow its contents to settle until the supernatant liquid is only slightly cloudy (it is normally sufficient to allow the contents to settle overnight).

Table — Density of water as a function of temperature between 15 and 30 °C

Temperature	Density
°C	g/cm <sup>3</sup>
15	0,999 099
16	0,998 943
17	0,998 774
18	0,998 595
19	0,998 405
20	0,998 203
21	0,997 992
22	0,997 770
23	0,997 538
24	0,997 296
25	0,997 044
26	0,996 783
27	0,996 512
28	0,996 232
29	0,995 944
30	0,995 646

**7.2.3** Carefully fill the pycnometer, insert the glass stopper and carefully eliminate the liquid that overflows. Put the pycnometer into the thermostatically controlled bath (5.4) and raise its temperature to between 2 and 5 °C above the ambient temperature (this temperature is the temperature of the test to which the whole determination is related). Maintain the temperature constant to within  $\pm 0,2$  K.

**7.2.4** As the temperature rises, a little liquid will escape from the capillary bore of the stopper. Carefully remove this overflowing liquid by absorbing it with filter paper. The pycnometer has attained the test temperature when no more liquid comes from the capillary bore. Take the pycnometer from the thermostatically controlled bath and take precautions that heat from the hand does not warm the pycnometer and cause any further escape of liquid (such warming can be prevented by plunging the completely filled pycnometer into cold water for a few seconds, avoiding wetting the top of the neck or the stopper). Carefully wipe and dry the outside of the pycnometer and weigh it to the nearest 0,000 2 g (mass  $m_2$ ).

### 7.3 Determination of the mass of the pycnometer filled with the liquid used

**7.3.1** Empty and clean the pycnometer and fill it almost completely with water or with the other chosen liquid.

**7.3.2** Repeat the procedure detailed in 7.2.3 and 7.2.4 so as to ascertain the mass of the pycnometer filled with the liquid used (mass  $m_3$ ).

## 8 Expression of results

**8.1** Calculate the true density  $\rho$  from the expression

$$\rho = \frac{m_1}{m_3 + m_1 - m_2} \times \rho_{\text{liq}}$$

where

$\rho_{\text{liq}}$  is the density of the liquid used at the temperature of the thermostatically controlled bath (for the density of water, see the table);

$m_1, m_2, m_3$  are the masses determined in accordance with clause 7.

**8.2** Express the true density in kilograms per cubic metre or grams per cubic centimetre to three places of decimals.

## 9 Test report

The test report shall include the following information :

- a) the testing establishment;
- b) the date of test;

- c) a reference to this International Standard, i.e. determination of true density in accordance with ISO 5018;
- d) the designation of the material tested (manufacturer or source, type, batch, number, etc.);
- e) if applicable, the number of test pieces per item (i.e. per brick);
- f) the mass of each sample;
- g) the grain size after grinding;
- h) the heat treatment, if any;
- j) the pressure of the vacuum used;
- k) the liquid used;
- m) the test temperature;
- n) the individual values and the mean value of the true density for each item.



## Annex

### Alternative procedure for materials that are difficult to wet

- A.1** The following alternative procedure may be adopted, in the place of the procedure specified in 7.2, if the test material is difficult to wet with the liquid and if, after subsequent addition, its suspension in the liquid is not very good.
- A.2** Pour into the dry pycnometer a quantity of deaired boiled water or of another liquid of known density, the pycnometer being filled to no more than 1/4 of its capacity. Weigh the pycnometer and liquid to the nearest 0,000 2 g.
- A.3** Introduce into the pycnometer an amount of dry test material equivalent to approximately 1/3 the pycnometer volume. Again weigh the pycnometer to the nearest 0,000 2 g.
- A.4** The difference between these two weighings is the initial mass of the test material,  $m_1$ .

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